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MICRO-FLUIDIC CHEMICAL REACTOR SYSTEMS: DEVELOPMENT, SCALE-UP AND DEMONSTRATION

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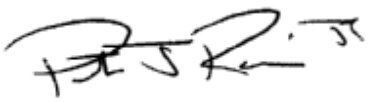
APPROVED: 

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13. ABSTRACT ***(Maximum 200 Words)***
This report summarized advances in microfluidic chemical reactor systems obtained using microfabrication techniques. Using these techniques, reduction in size and integration of multiple functions for chemical reactor systems were realized. Issues in combining microfluidic components, sensors and control elements into a micro chemical system were also explored.

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1 Accomplishments

The following sections describe research results obtained as part of the DARPA MicroFlumes program. Additional information is given in the paper listed in Section 2.

1.1 Gas-Phase Reactors for Wall Catalyzed Reactions

A common microreactor design used for gas-phase heterogeneous reactions is a microchannel device that can be integrated with a heat exchange layer for highly exothermic reactions.[6-1] These devices have the advantage of high productivity per unit volume, but they suffer from lack of sensing and active control within the microchannel assembly–similar to conventional ceramic monolith reactors. Thin walled microreactors offer the opportunity for the integration of heaters, as well as flow and temperature sensors on the external side of the reaction region (see Figure 1). The micron-thick wall provides good thermal contact with the catalyst in the interior, and the low thermal mass of the thin wall has the further advantage of fast (~10 ms) thermal response times.

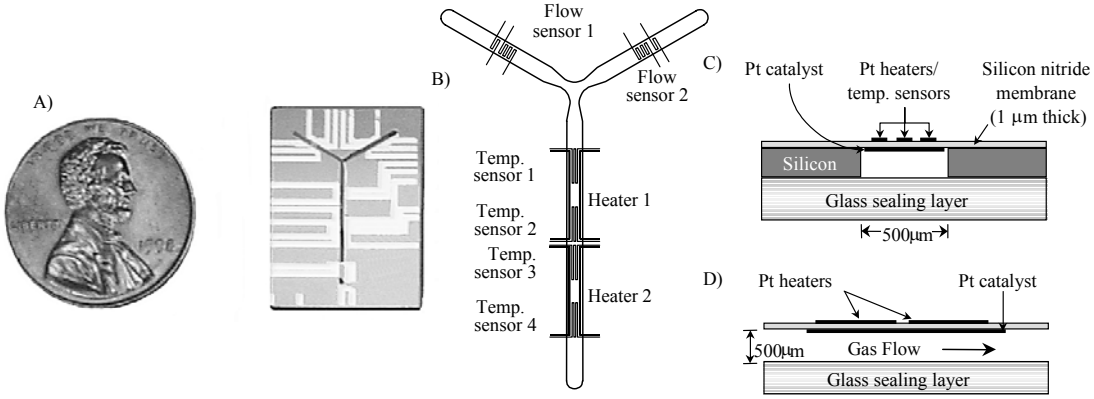

Figure 1: Gas Phase Microreactor. *(A) Photograph of device. (B) Top view schematic. (C) Side view across channel. (D) Side view along flow direction.*[2]

The catalyst can be deposited on the interior wall by a variety of techniques including thin film deposition by physical or chemical vapor deposition, and liquid preparation techniques. Thin film deposition techniques of the catalyst are typically limited to metals and simple oxides. Moreover, the resulting film has a low surface area and must be roughened by thermal activation. Wet preparation techniques allow the creation of catalyst systems similar to those used in conventional reactors, but surface tension effects cause the catalyst solution to collect in channel corners. Aerosol sprays or ink jet printing allows placement of the catalyst on a desired region of the membrane.

Oxidation and partial oxidation reactions of a number of compounds, including hydrogen, ammonia, and ethane, have been successfully carried out in the flammable regime. [3] The ability

[1] G. Wießmeier and D. Hönicke, "Microfabricated Components for Heterogeneously Catalysed Reactions", *J. Micromech. Microeng.*, *6,* 285-289 (1996).

[2] D. J. Quiram, K. F. Jensen, M. A. Schmidt, P. L. Mills, J. F. Ryley, and M. D. Wetzel. "Integrated Microchemical Systems: Opportunities for Process Design" in *Foundations of Computer Aided Process Design*. 1999. Colorado, CACHE, Colorado (1999),

to control the catalyst temperature and, therefore, the selectivity of partial oxidation reactions, is a critical factor determining reactor performance. During an exothermic reaction, the heat is supplied to the catalyst wall by both the heaters and the reaction. The primary heat loss mechanism is through conduction in the thin top wall to the reactor bulk silicon side walls. Therefore, by varying the thickness and thermal conductivity of the thin top, the ability of the reactor to dissipate the heat of the reaction can be controlled (see Figures 2a and b). This feature provides access to a wider temperature window of operating conditions and milder oxidation conditions than can be achieved in conventional systems. Alternatively, the device could be optimized to sense the energy released, and serve as a microcalorimeter.

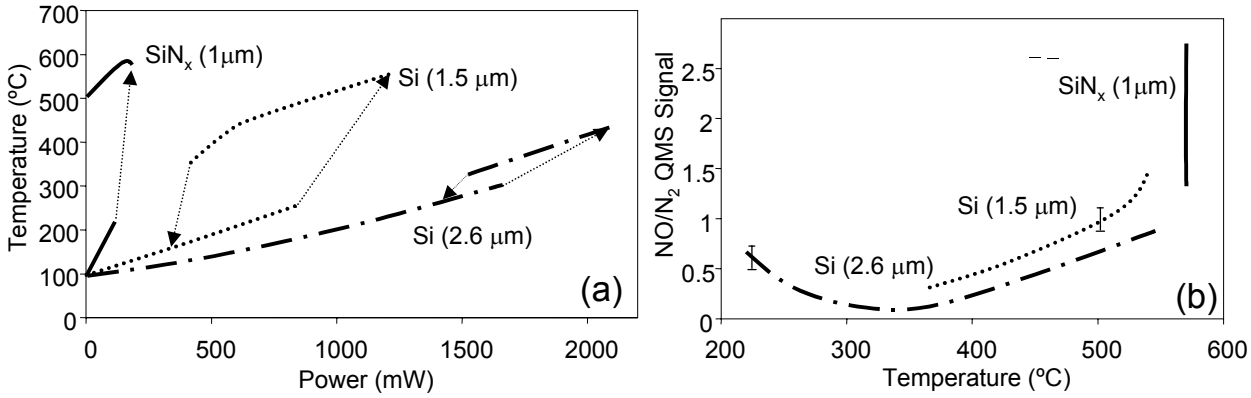

Figure 2: Ignition/Extinction Behavior and Selectivity *(a) Ignition/extinction behavior with varying input power for ammonia oxidation over platinum in microreactors with different wall materials and thickness. (b) Corresponding variation in selectivity towards nitrogen and nitrogen monoxide. Note, the greatly enhanced temperature window for controlling selectivity.*

Membrane Microreactors

The use of a permeable membrane allows the integration of separation with chemical reaction, as in macroscopic membrane reactors.[4] For example, the integration of a submicron-thick palladium membrane makes a highly efficient hydrogen purification device and provides the potential for conducting hydrogenation and dehydrogenation reactions.[5] Figure 3 shows an example of such a device, the ~200 nm thick palladium film is supported structurally by a composite silicon nitride and oxide thin film with 4 μm wide holes. This approach allows the use of other membrane materials for highly selective gas separation. Alternatively, the holes in the support structure can be used to disperse gas into a liquid stream, or can act as a particle filter.

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[3] R. Srinivasan, I.-M. Hsing, P. E. Berger, K. F. Jensen, S. L. Firebaugh, M. A. Schmidt, M. P. Harold, J. J. Lerou, and J. F. Ryley, "Micromachined Reactors for Catalytic Partial Oxidation Reactions", *AIChE Journal*, *43,* 3059 (1997).

[4] R. Govind and N. Itoh, "Membrane Reactor Technology", *AIChE Symposium Series*, 268, AIChE, New York, (1989).

[5] A. Franz, K. F. Jensen, and M. A. Schmidt. "Palladium Based Micromembranes for Hydrogen Separation and Hydrogenation/Dehydrogenation Reactions" in *12th International Conference on MicroElectro-Mechanical Systems*. Orlando, Florida, IEEE (1999), 382-385.

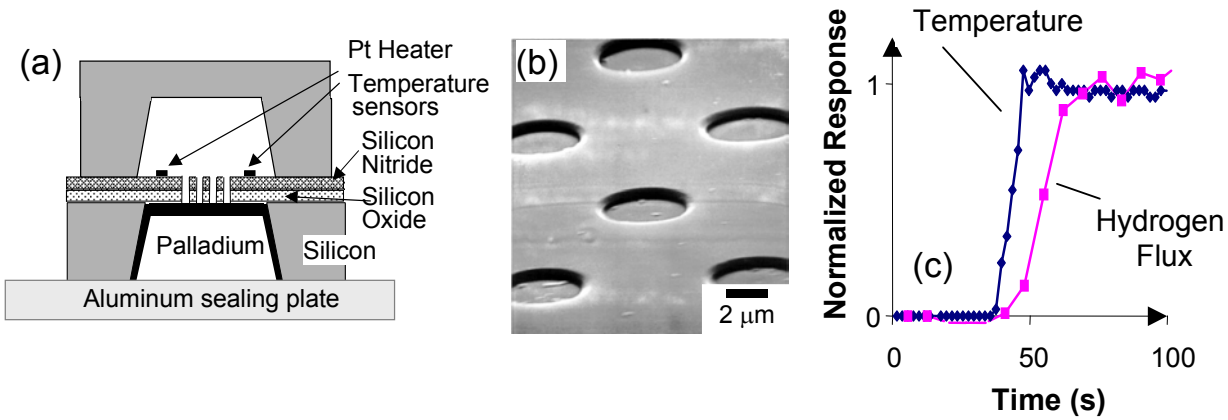

Figure 3: Palladium membrane microreactor. *(a) Schematic of cross-section perpendicular to flow (b) SEM of membrane - holes are 4 μm wide, 200 nm thick, free standing palladium films. (b) Thermal and hydrogen flux response of device - typical hydrogen fluxes are 600 std. cm^3/cm^2 for a Δp=0.1 atm.*

1.2 Liquid Phase Reaction Systems

Mixing is a critical issue in the design of liquid phase microreactors. The small dimensions in microfluidic devices imply small Reynolds numbers and laminar flow so that mixing occurs by diffusion. This characteristic becomes both a challenge and an advantage for liquid phase reaction systems. The relatively slow mixing can be exploited in phase transfer reactions and separation devices by co-flowing streams so that one (or more) reagents can be extracted from one fluid to the neighboring fluid.[6,7] Each stream can then be separated again into its components by splitting the channel. Surface tension differences among fluids and channel walls can be exploited to gain further control of the process.

To accelerate mixing, most liquid phase reaction systems rely on splitting and recombination of the fluid streams several times to create a laminated fluid with an increased fluid interface and shortened diffusion path.[8] The choice of design is a tradeoff between mixing speed, pressure drop, volume flow, and the feasibility of microfabrication. In all cases it is useful to be able to make high aspect ratio structures that produce thin layers of fluids. Figure 4 shows an example of a microfabricated liquid phase reactor that integrates laminar mixing, hydrodynamic focusing, rapid heat transfer, and temperature sensing. Deep reactive ion etching (DRIE)[9] was used to form channels 50-400 μm wide and ~500 μm deep. An anodically bonded Pyrex wafer capped the wafer. Model studies with acid-base reactions show that the reactor achieves complete mixing in ~10 ms, depending on the fluid properties.[10] The relatively high transfer coefficient

[6] J. P. Brody, P. Yager, R. E. Goldstein, and R. H. Austin, "Biotechnology at Low Reynolds Numbers", *Biophysical Journal*, *71,* 3430 (1996).

[7] J. R. Burns and C. Ramshaw, "Development of a Microreactor for Chemical Production", *Trans IChemE*, *77,* 206 (1999).

[8] W. Ehrfeld, K. Golbig, V. Hessel, H. Lowe, and T. Richter, "Characterization Of Mixing In Micromixers By a Test Reaction: Single Mixing Units and Mixer Arrays", *Ind. Eng. Chem. Research*, *38,* 1075 (1999).

[9] A. A. Ayon, R. Braff, C. C. Lin, H. H. Sawin, and M. A. Schmidt, "Characterization of a Time Multiplexed Inductively Coupled Plasma Etcher", *J.Electrochem. Soc.*, *146,* 339 (1999).

[10] T. M. Floyd, M. W. Losey, S. L. Firebaugh, K. F. Jensen, and M. A. Schmidt, "Novel liquid phase microreactors for safe production of hazardous specialty chemicals." In *3rd International Conference on Microreaction Technology,* Springer, Frankfurt (1999).

[1500 W/(m2·Cº)] realized in the microfabricated heat exchanger provided excellent cooling of even highly exothermic reactions such as hydrolysis of propionyl acid chloride.

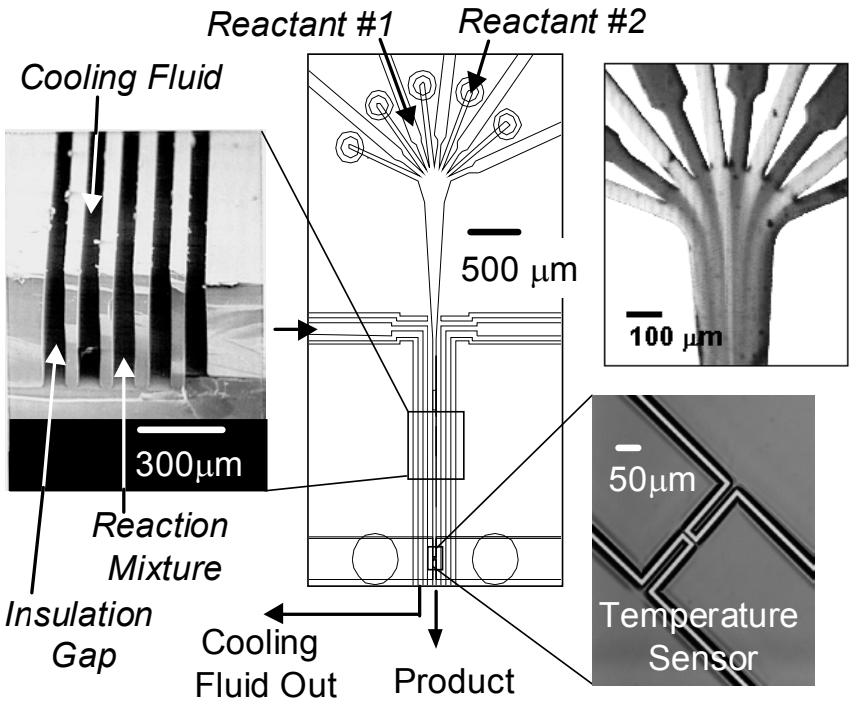

Figure 4: Liquid phase reactor with lamination of fluid streams. *Heat exchangers and temperature sensors in downstream reaction zone. Upper left-hand insert shows mixing of acid base mixture in gray tone image of indicator color.*[17]

1.3 Multiphase Microreactors

Gas-liquid-solid reactions are ubiquitous throughout the chemical industry and provide unique opportunities for microfabrication. The high surface-to-volume ratios attainable in microfabricated structures, leading to improved thermal management and fast mass transfer, suggest that microfabricated multiphase systems could have performance advantages relative to conventional macroscopic systems. We have demonstrated these characteristics for single and multichannel, multiphase packed bed reactors (Figure 5).[11,12] These were again fabricated using DRIE technology and bonding techniques. In addition to the microfluidics, thin-film resistive heaters and temperature sensors have been integrated with the device. Each reaction chamber holds porous catalyst particles in place by a microfabricated filter as reactants are fed continuously in a co-current fashion. The gas and liquid reactant streams are brought into contact by a series of interleaved, high-aspect ratio inlet channels (25 μm wide × 300 μm deep) designed to increase gas-liquid mass transfer. The catalyst restrainer is formed within the reaction channel by etching a series of 40 μm wide posts separated by 25 μm gaps.

[11] M. W. Losey, M. A. Schmidt, and K. F. Jensen ,"A micro packed-bed reactor for chemical synthesis." In *3rd International Conference on Microreaction Technology,* Springer, Frankfurt (1999).

[12] M. W. Losey, S. Isogai, M. A. Schmidt, and K. F. Jensen. "Microfabricated Devices for Multiphase Catalytic Processes", in *4h International Conference on Microreaction Technology*. 2000. Atlanta, GA, AIChE, New York (2000), 416-422.

Selected multiphase systems have been investigated. Oxidation reactions, exemplified by the oxidation of benzaldehyde, have been demonstrated in a single-channel reactor system. In this case, the inherent safety of a small reaction volume (4 μl) allows the reaction of pure oxygen and organic solvents at elevated temperatures. The hydrogenation of cyclohexene has been used to characterize the mass transfer coefficient of the multiple channel reactor using traditional reaction engineering analysis. Values of the mass transfer coefficient for the multiple channel microreactor have been determined to be two orders of magnitude larger than those reported for typical macroscopic reactors. Therefore, for reactions that operate in a mass-transfer limited regime, microreaction devices could be considerably smaller in volume and still maintain high productivity.

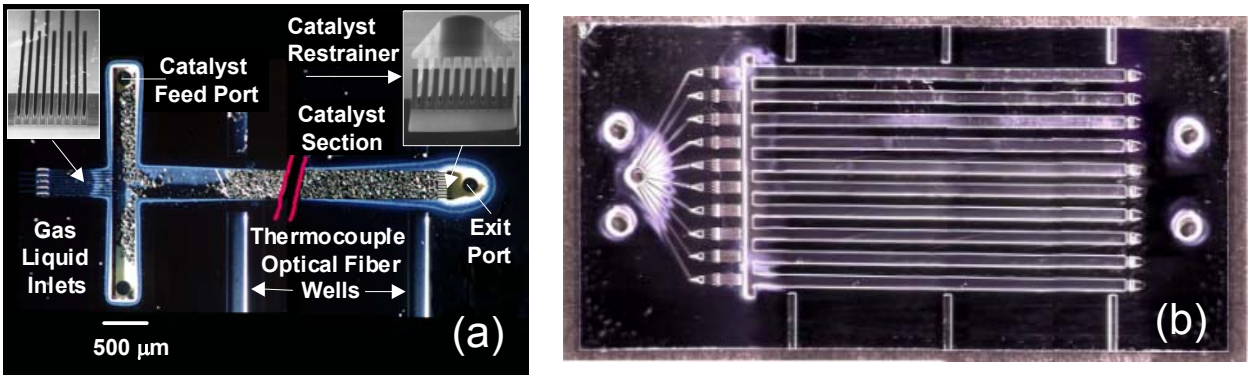

Figure 5: Multiphase Reactors (a) *Multiphase packed bed reactor with active carbon catalyst. (Left insert) SEM of gas-liquid inlet sections. (Right insert) SEM of catalyst support grid and exit on the right. Catalyst is introduced through the large wing sections.*[8] **(b)** *10 channel multiphase packed bed reactor.*

The reactor has also been used as a gas-solid packed bed reactor. The production of phosgene over activated carbon in this reactor exemplifies the use of the technology as an on-demand source of an important, but toxic, intermediate compound. The reaction is highly exothermic and reactants and products are difficult to handle. Complete conversion of chlorine to phosgene is achieved at 235°C for chlorine flow rates of 4 cm^3/min at standard conditions. At this rate, ~40 kg/year per device of phosgene production could be achieved in the 10 channel device. This production level suggests that useful amounts of phosgene for laboratory and pilot plant applications could be realized with a modest number of devices. The produced phosgene has also been used in on-line in reactions with liquid amines to form isocyanates.

1.4 Cross-Flow Microreactor for Kinetic Studies of Catalytic Processes

A microchemical reactor that utilizes a novel cross-flow geometry (Figure 5) and displays enhanced thermal control has been developed for the testing of porous supported catalysts. The cross-flow design reduces pressure drop by using a shallow but wide catalyst bed. 256 shallow-etched channels provide a pressure drop much larger than the pressure drop through the catalyst bed so that differences in catalyst packing density have negligible effect on the overall flow distribution.

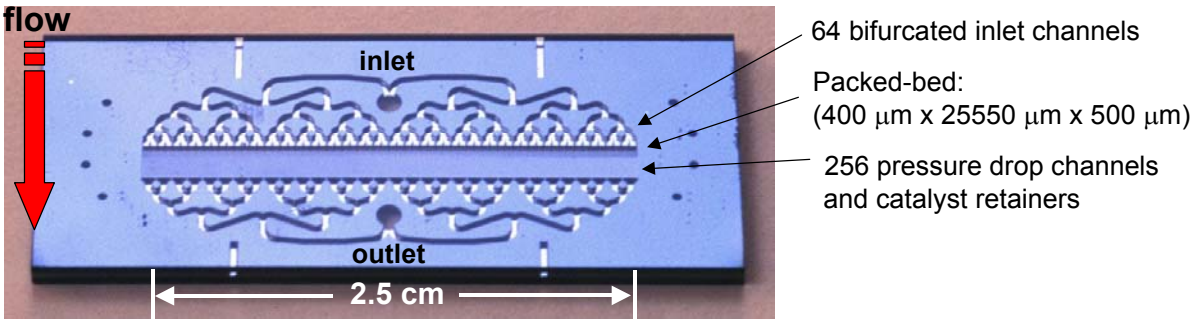

Figure 6: Silicon Microfabricated cross-flow reactor

The cross-flow microreactor has several advantages for testing heterogeneous catalysts. In addition to operating with differential conversions and minimizing pressure drop, the sub-millimeter dimensions enhance heat and mass transfer suppressing gradients in the catalyst bed. Commonly used macro-scale tube-in-furnace reactors are prone to thermal gradients, mass transfer limitations, poor temperature control, and large pressure drops that complicate kinetics extraction. Further, the cross-flow microreactor utilizes porous catalyst particles. This allows testing catalysts that closely resemble those used in industrial applications. Microfabrication technology also allows for parallel manufacture and operation needed for high-throughput catalyst testing applications. The reactor has been demonstrated in kinetic studies of model chemical systems, e.g., CO oxidation.

1.5 Microchemical Systems for Direct Fluorination of Aromatics

We have also developed an integrated microchemical system that enables efficient and safe direct fluorination of aromatic compounds, a process difficult to implement on a conventional macroscopic scale. The reactor (Figure 7), fabricated by using silicon processing microfabrication and metal deposition techniques, provides efficient gas-liquid contacting, selective fluorination, good heat transfer, and easy replication. The dimensions of the reactor were selected to maximize the area to volume ratio of the device and enhance the contact between fluorine and the substrate solution. Toluene was chosen as model chemistry because of past studies of the fluorination of this compound can serve as a benchmark. Conversions of toluene on the order of 80% were observed with no measurable temperature rise. The substitution pattern of the ortho, meta, and para isomers, determined via GC/MS, indicated that the reaction occurred by favoring the aromatic electrophilic mechanism.

1.6 Microreators for photochemical reactions

Performing photochemistry in microreactors has potential advantages over conventional setups. In industrial reactors, radicals generated by the UV radiation tend to accumulate near the light source. This may result in lower quantum efficiency and local overheating. In addition, crystal formation (often times on the light bulbs) during reactions not only makes cleaning after reaction difficult, but also hinders the propagation of photons. In contrast, microreactors could alleviate some of these problems by controlling the concentrations of the radicals through reactor design and controlling flows, and by removing heat adequately. A photochemical microreactor has been developed and tested using as model reaction the pinacol formation reaction of benzophenone in isopropanol (Figure8). The UV radiation source is a commercial miniaturized UV lamp.

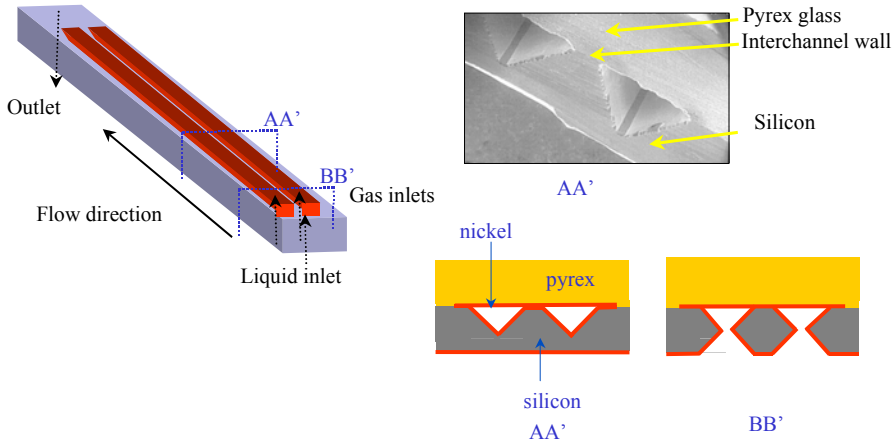

Figure 7: Microreactor Prototype Channel and Ports Cross Section

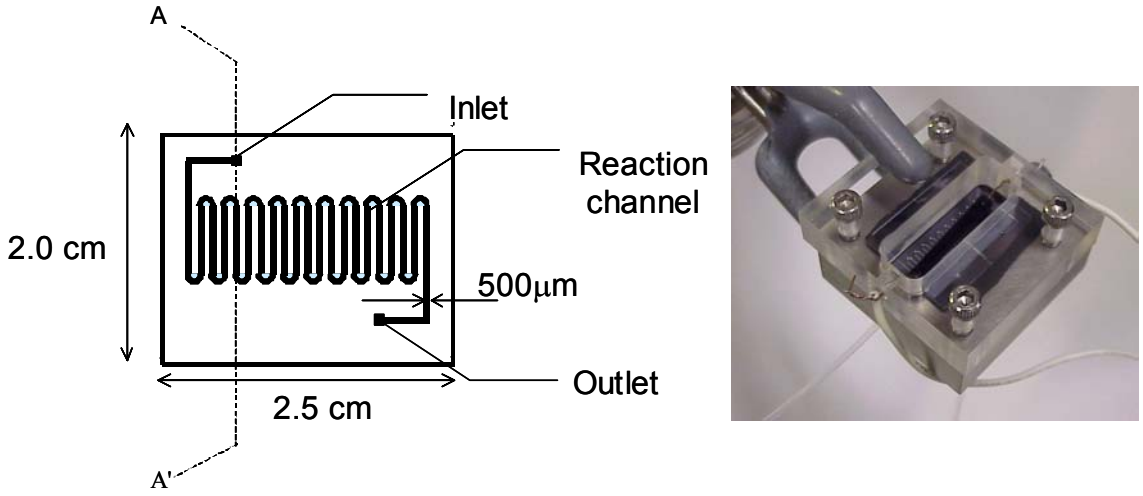

Figure 8: Photochemical Reactor

Electrochemical Microreactors for Electrosynthesis

Electrosynthesis has considerable promise for environmentally benign synthesis of organic compounds,[13,14] but its implementation has been limited by several factors. Diffusion of reactants through the gas or liquid layer adjacent to the electrodes typically limits productivity, and electrochemical reactors are more expensive than their standard catalytic counterparts. Poor current efficiency and the separation of products from electrolytes further add costs to electrosynthesis. Scaling an electrochemical reactor down to the microscale offers several

[13] P. M. Bersier, L. Carlsson and J. Bersier, "Electrochemistry for a Cleaner Environment," *Topics in Current Chemistry* **1994**, *170*, 114-229.
[14] D. Simonsson, "Electrochemistry for a cleaner environment," *Chemical Society Reviews* **1997**, *26*, 181-189

advantages in terms of enhanced mass transfer, electrode configuration, and controlled lamination of fluid streams. The initial electrochemical reactor is shown in Figure 9[15] – the reactor takes advantage of laminar flow to keep catholyte and anolyte separated. The structure is designed so that the catholyte and anolyte can be introduced into the system separately. Because the flow is laminar and because the channels have a low aspect ratio (~ 500 microns wide and 50 microns deep), the streams have enough time to react at the surface of the electrodes, but not enough time to mix.

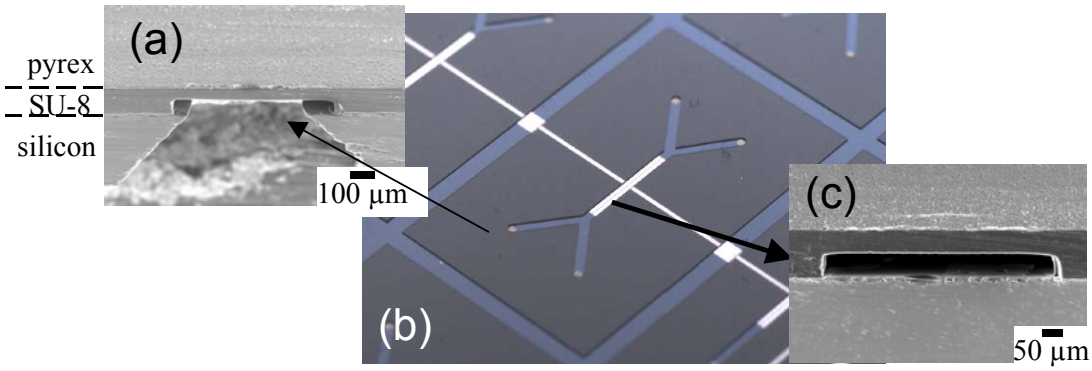

Figure 9: Photograph of Electrochemical Microreactor *(b) and scanning electron microscopy images showing cross-sections through the inlet (a) and channel (b). SU-8 is a photosensitive epoxy (discussed below).*

1.7 Integration of Chemical Sensors

Several of the devices illustrated above have included temperature sensors. For many applications, it is desirable to determine chemical composition and concentrations, which is typically a difficult and time-consuming task for macroscopic systems. In order for microreaction technology to be successful, it must include chemical sensors. Having a small reactor interfaced to large bench top analytical equipment often implies that external, fluid chamber volume is much larger than the volume of the microreactor. This discrepancy raises the potential that the observed reactor volume is the reactor-analytical system interface, instead of the microreactor. The problem is avoided only if (i) the reaction reaches full conversion in the microreactor, (ii) the reactants and products are separated in the microreactor, or (iii) the reaction is quenched in the microreactor.

In the case of liquid phase reactions that can be monitored by color changes or variations in the UV spectrum, the reactor can readily be interfaced to an optical fiber light source and detector to enable on-line monitoring (Figure 10). We have developed low temperature bonding methods that allow incorporation of quartz windows into microreactors for UV spectroscopy and photochemical reactions (see above). .

Infrared (IR) detection is a broadly applicable detection method that can be integrated with liquid phase microreactors by using the IR transparency of silicon. As an example, Figure 11 shows a small IR transparent T-reactor used to monitor the progress of the hydrolysis of an acyl

[15] J. J. Jackmann, T. Floyd, M. A. Schmidt, and K. F. Jensen. "Development of Methods for On-Line Chemical Detection With Liquid-Phase Microchemical Reactors Using Conventional and Unconventional Techniques" in *µTAS 2000 Workshop*. 2000. Twente, The Netherlands, Kluwer Academic Publishers, Dordrecht, The Netherlands (2000).

chloride.[16] Similar structures may be integrated with microreactors, such as those illustrated in the preceding sections. Thickness, doping of the wafer, as well as reflections from the substrate surface, adversely affect transmission. Although the 40% transmission obtained using uncoated samples is sufficient for most applications, one can apply an anti-reflective (AR) coating to increase silicon transmission.

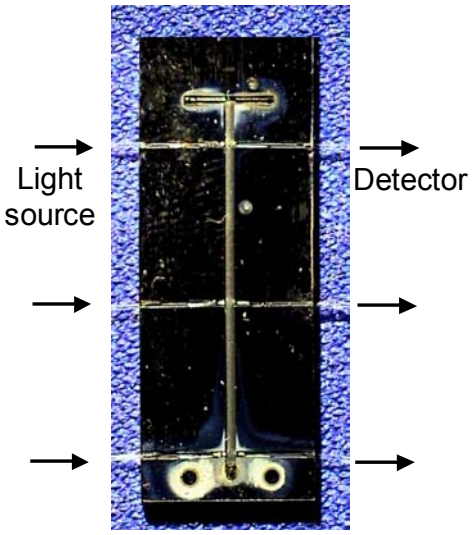

Figure 10: Microreactor with integrated optical fibers.

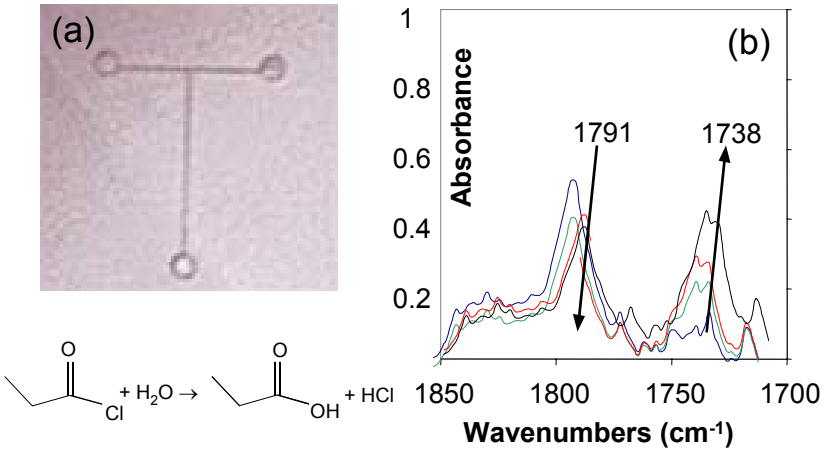

Figure 11: IR Transparent T-microreactor *(under IR illumination) (A) and IR spectra during hydrolysis of propionyl chloride (B).*

The short path lengths and optical densities usually imply that IR absorption spectroscopy is not practicable in gas-phase microreactors. As an alternative optical method for gas-phase detection, photoacoustic spectroscopy (PAS) offers several advantages. In photoacoustic spectroscopy, incident light is modulated at an acoustic frequency. If the optical wavelength couples to an energy transition in the gas, the gas absorbs the light resulting in a periodic gas expansion. PAS applies to many chemical compounds and its sensitivity scales inversely with dimensions. As a step towards a monolithically integrated system, we have microfabricated a photoacoustic detection cell (Figure 12a).[17] The cell was a 15 mm long, 700 µm wide cavity capped by a 2.3 µm thick silicon membrane. An optical fiber brought in the light of a 5 mW, 3.39 µm laser modulated with a chopper. The cell was capable of detecting propane in a background of CO_2 (Figure 12b).

[16] T. Floyd, M. A. Schmidt, and K. F. Jensen. "Towards Integration of Chemical Detection for Liquid Phase Microchannel Reactors", in *4th International Conference on Microreaction Technology*. 2000. Atlanta, GA, AIChE, New York (2000), 416-422.

[17] S. L. Firebaugh, K. F. Jensen, and M. A. Schmidt. "Miniaturization And Integration of Photoacoustic Detection With a Microfabricated Chemical Reactor System", in *µTAS 2000 Workshop*. 2000. Twente, The Netherlands, Kluwer Academic Publishers, Dordrecht, The Netherlands (2000).

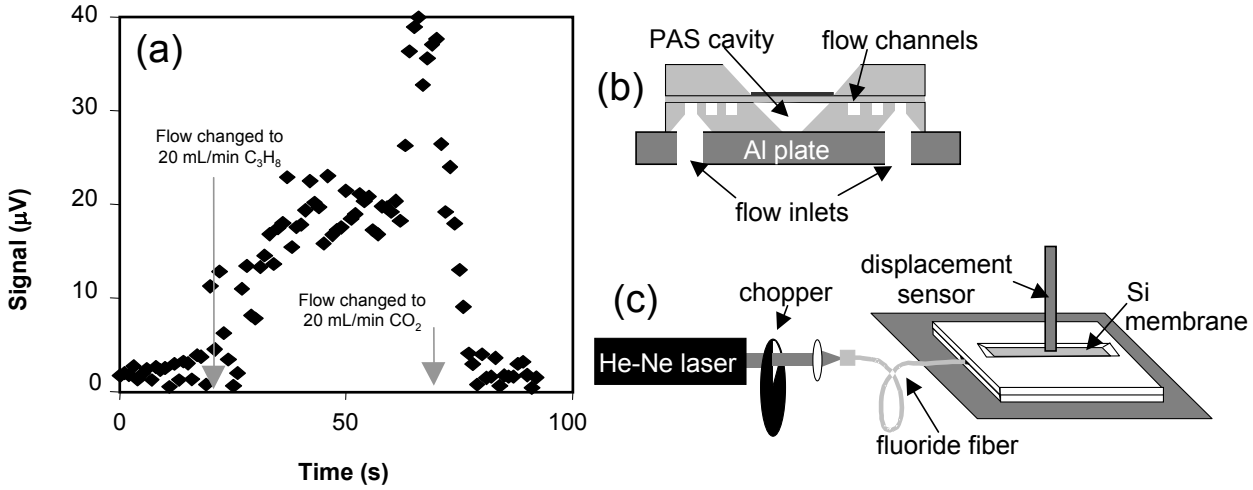

Figure 12: Photoacoustic Cell *Data (a) and schematic of microfabricated photoacoustic cell (b) and set-up (c).*

1.8 Combining MEMS and Soft Lithography

In many cases, silicon functions well as the substrate for a microchemical reactor, but different materials (*e.g.* polymers) that have desirable material properties, may be preferable for some application.[18] The MIT efforts have also explored alternative fabrication strategies for microreactors based on soft lithography[19] and electroplating approaches. We have been particularly interested in the photosensitive epoxy SU-8[20] since it offers opportunities for producing hybrid microchemical devices in silicon and SU-8, quartz and SU-8, and entirely from SU-8. In all cases silicon, quartz, or Pyrex has served as the substrate on which the device was formed. SU-8 has been used to create sealed microfluidic channels (see Figure 13) to bond materials and to planarize structures. The ability to planarize allows patterned metal structures (for electrochemical detection/synthesis or for heating) to be incorporated into reaction chambers. The flexibility creates additional opportunities for liquid-phase microreactors. At low temperatures, for example, it becomes feasible to fabricate composite quartz/SU-8 devices that allow for *in situ* UV detection through a quartz window.

1.9 Simulation of Microreactor Systems

An in-depth understanding of the operating characteristics of microreactors is necessary to evaluate benefits and disadvantages associated with new microreactor designs. Several commercial software tools are available to quantify different aspects of microfabricated devices, but they are not universally applicable. MEMCAD®[21] is an example of one popular software package for the design of MEMS devices. It includes the capability to model moving structures,

[18] R.J. Jackman, T.M. Floyd, R. Ghodssi, M.A. Schmidt, and K.F. Jensen, "Microfluidic systems with on-line UV detection fabricated in photodefinable epoxy," J. Micromechanical and Microengineering. *11* 263-279 (2001).
[19] Y. N. Xia and G. M. Whitesides, "Soft Lithography", *Ann.Rev. Mat. Sci*, *28,* 153 (1998).
[20] M. O. Heuschkel, L. Guerin, B. Buisson, D. Bertrand, and P. Renaud, "Buried Microchannels In Photopolymer For Delivering of Solutions To Neurons In a Network," *Sensors and Actuators B-Chemical*, *48,* 356 (1998).
[21] Microcosm Technologies, Inc., 5511 Capital Center Dr., Suite 104, Raleigh, NC 27606.

electric fields, heat transfer, and fluid flow in one package. It is moving towards the simulation of reactive flows, but this is difficult even for applications specifically designed for this purpose, such as CFD-ACE®[22] and Fluent.®[23]

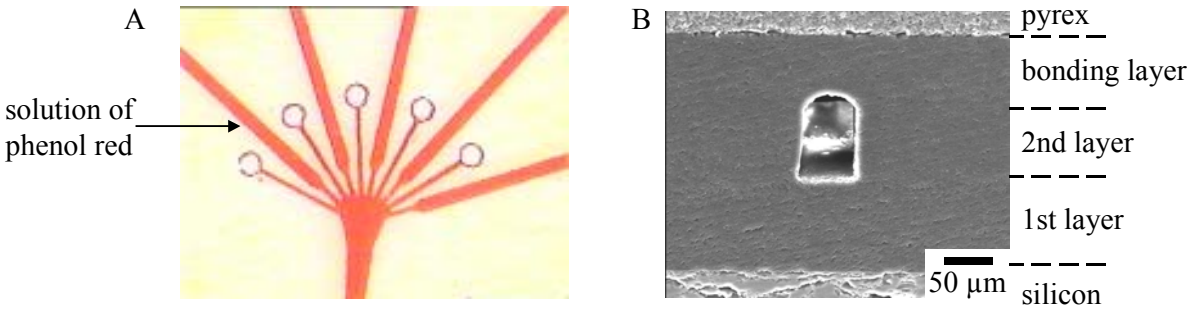

Figure 13: Micromixer device formed in SU-8 using SU-8 bonding technique. *(**A**) Photograph of a solution of phenol red in fluidic micromixer; (**B**) SEM image of a cross-section through SU-8 device (still mounted on silicon substrate).*

We have used simulation not only as a design tool, but also as a tool to assist in the interpretation of experimental data. Models have been used to evaluate design changes, such as changing the membrane material or the heater design,[24,25] and to predict mixing in liquid-phase reactors with quantitative agreement with experimental observations.[17] This computational approach to new designs avoids a costly, iterative experimental design process where components are fabricated, tested, and then redesigned to improve performance. In addition, the simulation tools serve in the interpretation of experimental data. For example, the simulations can clearly identify experimental regimes where mass transfer limitations dominate in determining reactor conversion. We will continue to integrate simulation tools with the experimental microfabrication effort so that the potential advantages of microreaction technology can be assessed.

1.10 Scale-Up

In the past, the microreaction community has focused on the design of individual microreactors. The concept of scale-up by replication of many units (scale-out) at first glance may appear to be simple, but the strategy presents new challenges that have not been addressed in previous chemical system scale-up. Particularly, the areas of reactor monitoring and control become increasingly complex as the parallel array size grows to a large number of reactors.

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[22] CFD Research Corporation, 215 Wynn Dr., Huntsville, AL 35805.
[23] Fluent Inc., 10 Cavendish Ct., Lebanon, NH 03766.
[24] I.-M. Hsing, R. Srinivasan, M. P. Harold, K. F. Jensen, and M. A. Schmidt, "Simulation of Micromachined Chemical Reactors for Heterogeneous Partial Oxidation Reactions," *Chem. Eng. Sci., 55*, 1 (2000).
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To address issues in microreactor scale-up, MIT and DuPont have constructed a multiple reactor test station for gas-phase systems.[26,27] The fabrication of this system serves as a test case for the development of more complex microreactor based systems. The approach addresses the difficulties in developing electrical and fluidic interfaces to microreactors that must be interchanged frequently, have heated outlet lines, and require millisecond real-time control algorithms. The multiple microreactor test station shown schematically in Figures 14 and 15 not only include microfabricated reactors, but will also integrate MEMS components for fluidic control. Such a system could ultimately contain all the components needed for a catalyst or process synthesis test station (feed gas manifold, reactor feed manifold, reactors, and control circuitry), but with a dramatically smaller footprint than current macroscopic laboratory equipment.

The joint effort with the DuPont Company work demonstrates the integration of multiple microreactors operating in parallel within a system that includes gas flow control components and the associated electronic circuitry. The system built is equivalent to a conventional laboratory reactor system but in 1/10th of the space. Fluidic and electronic interfaces, thermal management, and operational safety are all considerations in microreactor packaging. A standard microchip socket from Texas Instruments was selected as the first level packaging. The sockets have mechanical attributes that lend them to, with minor reworking, simultaneous fluidic and electronic connection. This selection makes PC board mounting of the reactor devices straightforward. Shut-off microvalves and proportional microvalves from Redwood Microsystems, with their control electronics, have also been mounted on PC boards to control the gas flow in the system. This allows the entire system: reactors, device electronics, and gas distribution manifold to be mounted on standard CompactPCI cards and housed in a commercially available chassis. A Kaparel CompactPCI chassis is used to house the microreactor system. Electrical connections between the boards are achieved through a standard backplane and custom-built rear I/O PC boards. The system is comprised of a temperature controller card that regulates temperature for auxiliary heaters in the system; a gas mixing board that mixes the feed gas for the microreactors; two microreactor boards that each contain two microreactors with their feed flow controllers; and two heater circuit boards that provide power to the microreactor heaters. A National Instruments embedded real time processor is used to provide closed-loop control and monitor system alarms. A host PC, running LabVIEW 6, is used as the human machine interface for operator interaction and historical data logging.

[26]D. J. Quiram, J. F. R. Patrick L. Mills , M. D. Wetzel, J. M. Ashmead, T. M. Delaney, D. J. Kraus, J. S. McCracken, K. F. Jensen, and M. A. Schmidt. "Package Level Integration of Silicon Microfabricated Reactors to Form a Miniature Reactor Test System", in *Technical Digest of the 2000 Solid-State Sensor and Actuator Workshop*. 2000. Hilton Head Isl., SC, Transducer Research Foundation (2000).

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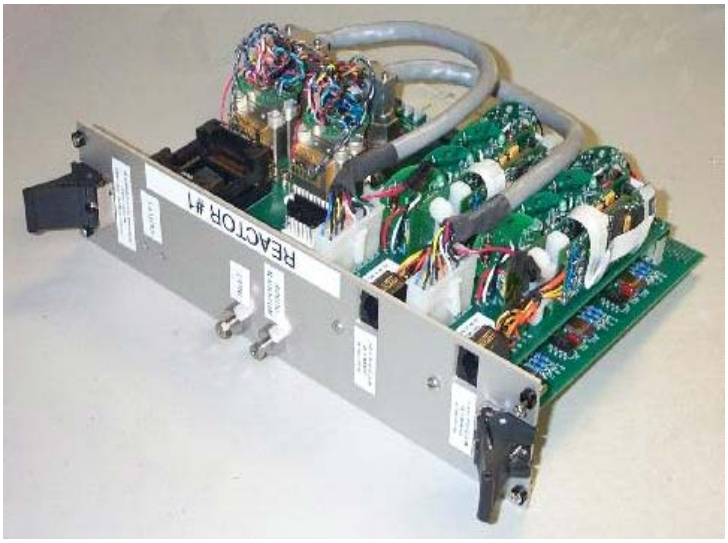

Figure 14: Microreactor electrical and fluidic card showing the integration of MEMS fluidic elements with microreactors.[30]

Figure 15: Microreactor system showing controller, heater power, fluid mixing, and microreactor cards (as in Figure 14) in a PCI chassis.[30]

2 Publications from the MIT Microreactor Group

2.1 Theses

1. Ravi Srinivasan,"Microfabricated Reactors for Partial Oxidation Reactions," Ph.D. Thesis, Massachusetts Institute of Technology (1998)
2. I-Ming Hsing,"Simulation Strategies for Microfabricated Chemical Systems," Ph.D. Thesis, Massachusetts Institute of Technology (1998)
3. Matthew W. Losey,˝"Novel Multiphase Chemical Reaction Systems Enabled by Microfabrication Technology," ," Ph.D. Thesis, Massachusetts Institute of Technology (2001).
4. Tamara M. Floyd, "A Novel Microchemical System for Rapid Liquid-Liquid Chemistry," Ph.D. Thesis, Massachusetts Institute of Technology (20001).
5. Samara L. Firebaugh,"Miniaturization and Integration of Photoacoustic Detection," ," Ph.D. Thesis, Massachusetts Institute of Technology (2001).
6. David J. Quiram," Characterization and Systems Integration of Microreactors ," Ph.D. Thesis, Massachusetts Institute of Technology (2001)
7. Sameer Ajmera, "Microchemical Systems for Kinetic Studies of Catalytic Processes," Ph.D. Thesis, Massachusetts Institute of Technology (2002)

2.2 Papers

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